Notebook Series -- Notebooks by Edison
Notebook, N-18-11-26

This notebook was used by Edison during November-December 1918. The entries pertain to experiments with primary batteries, battery components, and the processing of lithium ores. The notes in the first part of the book relate to the construction and performance of twelve experimental cells made with CuO electrode plates of varying types. These experiments are continued in N-18-12-26.1. The performance tests include measurement of amps, as well as tests of fragility by dropping plates. Subsequent entries relate to experiments on processing lithium ores involving lepidolite (lithium mica) and amblygonite. Reagents and procedures are given for each process, along with notations of the presence of a lithium "line," as indicated by a spectroscope. The lithium experiments are continued in N-18-12-26.2. Also included is a list of organic solutions in which Edison tested the solubility of lithium chloride, along with notes of results from the tests described in N-18-12-21 [not selected]. The lithium experiments probably pertain to the use of LiOH in the electrolyte for batteries. There are also cursory descriptions of experimental cells numbered from 13 to 19. The notes indicate that Frederick P. Ott assisted Edison with the battery experiments, while Ludwig F. (Louis) Ott assisted with the lithium experiments. The front cover is labeled "CuO Battery Expts & Lithia." The pages are unnumbered. Approximately 120 pages have been used.

Note ↓

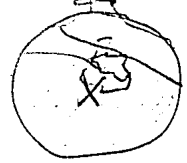
The Silicate Na ~~[illegible]~~ made by pouring a piece together with fingers, a dried half plate, put in NaOH 20%
Nos 22 was OK Dec 6th but on 16th had all disintegrated & gone to powder showing NaSilicate is no —

The Copper after reduction which was perfect all the way there was not several but perfect mechanically but was brittle & would not stand shipping without breaking in pieces —

Nov 26 1918 —

No 1 fruit jar experiment test 6 cells
5 grms NaSilicate, 100 grms Red Cu Silver Lake powder, pressed 800 lbs on gauge, Lab pieces Equals Thick, only Need lbs for in-
Coppered by melting dry Copper flour on by hand & dropping

No 1 Has broken plate

Na —  only the X

Part was coppered. Dried on hot plate only

No 1 Cell —

all reduced perfect

A part N fell out, Took out Copper found breaks very easy yet Except Center Could handle OK but was intact, gave more than Reg plates per gram made by Silver Lake —

No 2 Battery first jar is

5% Silicate Na but Coppered by Zinc dust rubbed on finger then 10% H_2SO_4 water by finger till all covered then washed & assembled

This is a whole plate & fairly well Coppered,

Its swelled bad in Cell probably due to H_2SO_4 going in pores –

Must be wet with water first & smallest amount acid possible be used so none penetrate, if Na Silicate is to be used, – possibly water should be a little alkaline

5 grams Na Silicate 100 grams CuO

500 lbs gauge pressure = 1000 lbs per inch? —

based on last photo for complete known solution,
20 drops on bottom pipe
10 inches high – 4 drops
at 18 inches –
This is merely an idea based
on the Reg 1900 formula based
CuO from Edgar Leche –
at 10 grams Eureka –

Do for Sulphide Sodium Ferric
Hydroxide Borax – break
on Hemet Lake.
Borax Cracks in Cakes

3 grams Na Silicate 100 CuO
breaks 1st drop at 10 inches
needs 5 grams –

About 10% clay - 100 CuO
pressed - dried hot plate
then in oven to Dull Red

broke 1 drop but clean
Crack center, no powder
10" drop, needs Fuller
higher broke or 15 grams
Clay -

Zinc hydroxide, too much water in it = a Red heat stood 1 10" drops make only fair.

If you use clay or brat to red you would have trouble to use acclimed Copper, same with Zinc Ox Hence this is not Commenced on the other hand

Returned plates with Silicate soda could be boiled with NaOH & Silicate Eliminated

Boric & Phos acid would not be objectionable if they dont go to process NaOH Cell —

Our $2\frac{1}{4}$" circular plate of CuO is 1/10th surface of the Reg Leclanche plate, 1 amp discharge rate is 200 Ml amp

3 amp rate 600 Ml amp

I evaporated down NaOH saturated with Zinc from Examination of new $Zn^{+}CuO$ when poured out got solid & strong - but it very soft in 20% NaOH Therefore N.G.

Dry Sulphate of Alumina.

Aluminate of Soda.

Phosphoric acid

Boric acid.

5 grams Ammonia Carbonate
Dissolved 6 cc H_2O
baked hot plate —
1 Drop - ~~[illegible]~~ no powder
12"

6 cc Ammonia
Baked . very soft powder
12" Drop
Neg

10 grams Oxide Zinc Hydrox
1 Drop 12" now powder
not promising

Took Shake Red ox - 100 grms
3 grms solid NaOH, dried on
plate, then for 1 hour kept
in Muffle at dull red
It broke on 2nd drop
no powder —
But it only 1/2 burnt
air does not circulate
+ Red oxide needs air
to be burned in a dense
plate. The Red showed
the beginning to black
whole in powder to
done very rapidly at
+ below a red heat,

Then goes black far below red

Red should have all the large pieces screened out as these burn to black very slowly compared to the fine —

So far ideal is to burn red in powder in presence of plenty air while powder is kept in motion —

The locked together with silicate soda. 5 parts to 100 of oxide dried around 300 deg take for 3 or 4 hours 4000 lbs pressure

Lake Red ox has about
10% left on 60 mesh
This is entirely too Coarse
to oxidize quickly & should
be taken out, Yet it is
practically all the
metallic copper —

A shaking screen will
take it out fine & it
can be recovered &
go thru process again

At SL they grind up old
plates seperate the lead
white Zinc mercadeon
then treat powder with
Barium Nitrate water
& use as new

Fred Ott made a desc of
Red oxide + 5 pts
Silicate Na to 100
CuO - Dried hot
plate, cant break by
drop on hard board
Exceedingly strong

It is very likely if you
put all our Red oxide
thru 80 to 100 mesh
it will oxide very rapid
& plate will give higher

Voltage as the surface exposed will be from 2 to 3 times the area

Made 3 2¼ discs
422/1000 thick

Used 100 grams Sd CuO &
5 grams Silicate Soda as
bought no water,

4000 lbs sq inch pressure

dried 1 hour hot plate 275

Fahr —

Shrinkage in thickness
003 - average
011 in Diameter

There is practically no shrinkage

Diameter of Bore are
precisely 2.288/1000

Lake black CuO produced
passed thru 200 mesh
shows quite an amount
of Red Cuprous

Notice it (16) CuO very
easily pulverized —

think we can raise
density up to near theoretical
very easily to 95%

Nov 29 1918

I find that by screening out the Coarse from Iron turnings (Cast) that it can replace Zinc powder for coating the CuO plates with Copper —

Using Red ox Iron 60 mesh & butter dish over 1/8 of sand on top of asbestos gauze & Bunsen burner fired all in 1 hours got whole black at 7 pm but on grinding found it only went in a little

Each 500 amp plate weighs about 2 lbs —

870 gms

Theoretically should get 680 amp h — ~~at 1 amp rate~~ get 438 at 3 amp or 581 at 1 amp rate

85.4% at 1 amp 70 Fahr

64.4 at 3 amp

Zinc Efficiency should give 810 amp at 1 amp rate get 593 — 73% Efficiency

NaOH, 79% Efficiency

Fred Ott now made a 1/32 steel plate & will try it over again — Temp around 700. 1/2 heavy black but dark red after grinding

Way want 2 or 3 hours —

Started 5.15. powdered as above

after 3 hours on powdering it still shows reddish — Temp is about 750° Fahr

It cannot be made CuO at this temp. in 3 hours at the size grains are — it seems there to be that size penetration where the CuO to the inner core of Cu_2O is exceedingly slow — Must have higher temp to hasten it.

Reg 500 amp plate
HL — 43 mlq per
gram of CuO plate is
amount of absorption
of water. LCtt. —

Wt before soak	125.630
" after "	131.120
	5.490

Our 2.25 Disc
100 CuO, 5 Na Acetate
Baked on hot plate
4 hours

Wt before soak	101.980	9.17% water
after "	111.280	
Water absorbed	9.300	

.091 per gram of oxide
is water absorbed
or over double Reg plate

Phos Soda goes topsiu

Aluminate of Soda evap
down on butter dish
36 hours — got very
hard, tough, 24 hours
in air still dry & hard
but it all dissolved
in water in 1/2 hour
U q for Cu

all baked dull red

3 clay + 3 clay with more water 1st drop break 1st drop - not strong enough,

5 clay thick + 5 clay with more water - only 1 drop —

5% clay not enough - want probably 7 to 10% clay

3 clay + 3 cc clay break 1st drop - 4 pieces

5 clay - 5 cc water.
break 2 pieces 1st drop
piece X stood several drops

Evidently 7 clay with 7 cc water will be OK

Silicate Soda dried in Butter dish 36 hours on hot plate
Dec 2 10 am put water over it renewed water Dec 3 10 am
at 3 o'clock Dec 3 all
the silicate had dissolved into the water ——

Hacks Book says its insoluble in K & Na salts
it looks that way. but I think 20% NaOH softens surface some & ultimately will make a mucilage
The duplicate dish with NaO has on 3rd 3 o'clock only slightly softened the surface

Conc acetates of Ca Mg Al are no good for CuO plate

Delicate of soda in
butter dish - on hot
plate for 36 hours —
taken off & water put
on, in 2 days water
had dissolved considerable
renewed water, in
1 day more the whole
had dissolved forming
a thick water glass
solution, This settles
it that NaSilicate is
too dangerous for a gen
immersion in 20% NaOH
although it appears not to

discolors in it after
4 days –

Powdered Lead glass
thru 200 mesh 6 grms
100 CuO 2 grms Caustic
Soda – Yellowish Red
heat
Stands 20 drops 1 ft
" 5 " 3 ft
on pad – also 5 drops
5 ft on wood floor
forced it down on 6th
& broke 2 pieces

Note, Make a Dese using water only + bring to yellow white & get porosity -

It coppers well with H_2SO_4 - 10% + Zinc dust Edges sharp -

Porosity - Weight before water 108,620 grms

Weight after immersion 115.900.

.067 grms water per gram of CuO -

Reg Silver Lake takes up on .047 per gram of CuO hence glass is better,

100 CuO 10 grams plaster
Paris - 12 cc water
goes to pieces in 2 hours
in 20% NaOH.

Dec 4 1918

100 CuO - 5 grams
Lead glass Horne 200 mesh
water only (Baked yellow)
Break on the drop
1 ft high - another one
break 7/10 drop -
.078 Mdg water absorbed
per gram. The one with
2 grams NaOH Conc + 6 glass
gives .067 shows that possibly very
little changed by the soda

gave 60 mah to .5

or 522 amp Hours

for Reg 870 gram

Silver Lake Plate,

70° Deg fahr about

average temp —

after soaking 2 hours

washed off & dried on

hot plate, broke on

4th Drop 1 ft high

This is satisfactory

No 3 Cell.

100 CuO — 5 grams Lead

glass 1 gram Conc. Sol

NaOH, — Baked yellow red

20 Drops 1 ft

10 Drops 3 ft (on pad —

Didnt break. Dec 4 1915

This is now on test in

a Cell & Marked No 3

gave 57. at .57 Volts

next reading 24 hours after was

down — probably

I fused (yellow red) powdered glass 25 grams with 5 grms Concentrated NaOH- & found it all fused & hard, I could stand 1/2 the soda & be fused well Enough I think

5 grams powdered lead glass, + only water, baked yellow red —

Absorption of water .091 per gram of CuO —

Weight before immersion 100.820

after 110 ood

No 2 Cell which is the same, has swelled badly & is NG — But No 1 which is also the same is not swelled in 10 days so I think a Dep. & can't say why it shouldn't swell, perhaps baked on hot plate longer However Nadhcate isn't promising — as compared to Either glass & Water or glass & same NaOH.

Disc Cupric ammonium + NaOH
8 days in NaOH 20%
all soft — NG

100 CuO 4 grms lead glaze.

500 Milg Dry soda in 5cc water
Baked Yellow red -
20 Drops at 1 ft } on pan
10 " at 3 ft }

Did not break, has a crack across

Absorption —— .073 per gram of CuO

on test Dec 5 1918

Wet before immersion	105.900
after	113.680
	7.780

Dec 5 1918

CuO 100 3 grms powdered glass
200 mesh –
250 Mil of dry soda + 5 cc water
Baked yellow heat,

20 Drops 1 ft
1 Drop 3 ft

Absorbed —— 078 per gram of CuO

on test Dec 5 6 pm 1918

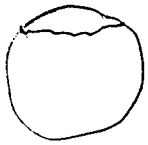
Weight before immersion 104.600
after 112 800
8.200

DEC 5 1918

100 CuO 2 grams Lead glass

160 Mlg Dry soda
5 cc water

2 Drops at 1 ft brake



Made no absorption test
with these in oven was a
mix of 5 Lead glass 1 gram
NaOH, — Dried on hot plate
it swelled up blebbly
but all fused showing
that much less soda
can be used.

Dec 6 1918

Portland Cement 10
CuO 100 — 4 days in
water — Dried —
1 Drop 1 ft broke in
5 pieces — N.G.

Fired OK made —
2 Discs of 100 CuO.
4 grms Lead of Cass Iron
200 mesh — 250 Mlg of
Dry Caustic Soda
Will put this in NO4 Cell
Soda did 5 cc water
One went 20 1st drops +
& 3 ft drops Dec 6.
put the broken disc in NaOH 20% for time test
over
5 pm

After run was over
Soaked 3 hours water —
dried — It broke at
3rd Drop, 1 ft

This is strong enough
a Salvagabony
Gives 5% amphoter

gave 58 amphours

Think this would be OK
& Satisfactory

~~Broke~~

Broke 4th time 1 ft
This would be satisfactory
gave 58 amp hours

Another 2 descs made
Same as No 4 but used
only 125 mlg Dry
Soda dissolved in 5 cc
water,

One of these went 20
drops at 1 ft & 3 drops
at 3 ft –
This will be No 5
Cell – Dec 6th
broken and put in 20% Soda for time test 5 pm
1 after run soaked 4 hours water, Dried on hot plate
Broke on 2nd Drop 1 ft –

Another pair with 62 mlg
Dry Soda 5 cc H_2O
this stood 20 drops at
1 ft + 2 drops at 3 ft
this will be No 6
Cell Dec 6
broken and put in 20% for time test 5 pm

Fred also made a thin disc made entirely of water & 200 mesh glass on iron plate — it only swelled up a little under micro its bubbly bubbles about 005 + clear all thru & perfectly fused together —

Dup of above with 250 mlg Dry Soda + 5 cc water - bright yellow. This swelled up more than ~~as much as~~ without soda but think it OK — fusion perfect, swelled 10% more But think his heat was too high considerable more than previous Experiments, It is fused clearer & much better than the above — 62 Soda which makes a strong Disc will probably be enough soda —

No 7 Cell-

100 CuO- thru 60 mesh
250 Mlg dry soda, 4 grams ground glass
thru 200 mesh, 5 cc water
10 drops 1 ft. [Note think was a flaw]
Baked higher than previous ones.
seemed at full yellow-

Made 1 drop at 1 ft- broke

This is OK but ought to drop
2 or 3 times to be perfectly
satisfactory -
Probably something wrong
in mixing -

Gave 61.95 amp hours to .5

Cell No 8.

100 CuO thru 100 mesh.

4 grams lead glass thru 200 mesh

250 mlg dry Soda 5 cc water

Baked full yellow

20 drops 1 ft 9 drops 3 ft

grid 63.45 avg hours. to .5

Broke 1st drop 1 ft no fuse

Think this will be

a duplicate

		Total amp H
	660	
	650	
	650	
	640	
	640	
	630	
	630	
	610	
	600	
10 days 24 hrs	600	52.350
	580	
	580	
	580	
	560	
	540	
	530	
	500	64.150
	258	

Cell No 9

100 CuO thru 180 mesh

4 powdered lead glass thru 200

250 mlg dry soda 5 cc water

Baked full yellow

20 drops 1 ft 7 drops 3 ft

This disc at 200 milamp discharge rate averaging 70 deg Fahr -

gave 64.150 amp hours

Equivalent of 558.1 amp Hours for large plate.

Broke at 2nd drop 1 ft but it had a flaw

This is OK + calculation

Thinking that last 2 bakes
Cells 5 + 6 & 7 8 + 9
were baked too high &
duplicated them & baked
so I could see thru the prep
hole that all were red
at dull red the edges were
black, but when turned up
edges were seen to be
bright & not black & ~~[illegible]~~
slapped heat

~~[illegible]~~ Cells

~~[illegible]~~

The glass recorder alone
with 250 mlg dry soda
4 grams glucose moistened
with water made into
dough & baked with
10 11 & 12. swelled very little
debut has glassy surface
but was well fused
together

OVER

Discs of this Cake described on preceding page.

Only gives 1 Drop at 1 ft & are entirely too weak.

The Experiment discs must Bake a bright Yellow to give proper strength & the disc of glass must have a shining or glassy surface —

Above were Reg SL Cu not screened which has very little strength compared to 100 mesh

Repeat (not screened) 100 CuO
Made one with 3 Lead glass
64 mlq Soda 5 cc aq
Baked yellow Red -
Broke 5th drop 1 ft
probably not high Enough
in heat or not Enough
soda - will Rebake
to higher temp, the
other one not broken
After baking full
yellow, broke on
5th 1 ft drop -
We must use CuO thru
100 mesh to get 3 Lead
glass to work -

Req 5 L CuO not screened
100 CuO | 4 grams BEST
Bottle glass, 64 Mulgdada
5 cc dq — same lamp
as on previous page
yellowish Red —
5 Drops 1 ft
probably not high
enough temperature
or not enough soda
will rebake the other
one at higher temp
After baking to full yellow
gave 20 drops 1 ft
2 " 3 ft
looks like 4 glass 125 Soda
+ 100 mesh CuO + yellow Red

Put remaining one in Cell & Mark it No 12

Note, it was cooled slowly

I had two of the 3 which broke 3rd drop - put back in Muffle & heated full yellow it made 20 1 ft drops + 7 3 ft drops & one 3 ft drop on floor

This is OK for bottle glass the heat must be higher than lead glass,

Make 3 Dies of 4 bottle glass 100 CuO thru 100 mesh 125 mlg Soda .5 cc of Yellow Red

+ 3 Dies of full yellow CuO thru 100 mesh - 125 mlg Soda 100 CuO, 4 bottle glass - 200 mesh

1st Lot - just yellow red only little red,

All broke 1st 1 ft drop second pieces Easily breaks with hand, bad mixing or something wrong as we have had with lead glass at Even a lower heat 20 drops 1 ft

see previous page,

Duplicated at ~~[illegible]~~ more yellow red & cooled slowly but broke 3rd drop 1 ft

No 12 Cell in bottle glass 4 -
with 125 mg Soda -
Heat was yellow red broke 3rd 1 ft
drop - put it back in furnace
& heat to full yellow
made 20 1 ft 7 3 ft -
& slowly cooled down
Want get capacity & voltage
This time diminished in
Dia .004 only

Broke 1st Drop 1 ft

But its OK

~~Made~~ The full yellow
ones well test on 11th
in morning

These came out OK 20 drops 1 ft
2 at 3 ft -

We let these cool down in
furnace. Think all cooling
should be slow otherwise
particles will be
unannealed & brittle
shattering on dropping &
perhaps on cooling too quick

Also they should be given
a low heat to cause
water to slowly evaporate
out & not make bubbles &
disrupt.

Should say 200 mg
Dry soda to 4 beer glass
200 mesh 5 cc H_2O should
be used to give margin
for variation of thermometers

Li_3PO_4

5.56287 phos Li corresponds to 1 gram Li in ore

perhaps 250 mlq soda could be safe ~

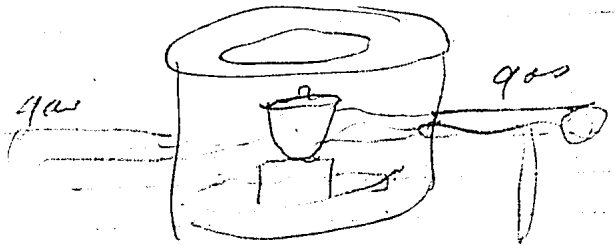
Put up a Cell — No 10

2 of this

Make some 100 mcd CuO
4 lead oxide 125 mg Boric
5cc water. When grinding or
rolling mixture of add 1 gram
of fine graphite & mix well —
Burn then bake yellow heat
+ Copper + put in CoCC
+ crack one —

Cell No 11

Lepidolite Dec 11 1915



Small Crucible holding furnace —
Use 1" x 2 deep graphite Crucible, 10 grms Lep 5 grms salt, 10 cc water
Evap off water by heat + when all off put in furnace heat to yellow heat, rod would show down easy the viscous filled mass (not liquid) took out / took cover off Lets make put it quickly back

Lepidolite wasn't moved short long 1/2 1/2

any furnace that melts it must have baffles at chimney to divert the volatilized stuff presumably chlorides —

After Crucible in boiling water for 1 hour Lep dont soften — Broke Crucible and got out mass & put in water — wont dissolve —

I exhausted it after powdered up, boiling water
hardly attacked by phosphoric after precipitating by excess NaOH

The put residue in 50% H_2SO_4 & heat to decompose remove H_2SO_4 —
This apparently does the trick
after cooling added water & filtered, added excess NaOH, some precip came down filtered it off — added NaPhos very large precip, small number fully ppt had change to another & larger

~~[illegible]~~

large breaking - started filtering found had not added enough phosphate, finally added 1/2 bottle full — [Too weak of quench]

Am now filtering - if this is phosphate this quench is going to be easy

10 Lep
10 plaster Paris —
Yellow white in furnace — not legured but soft viscous —

10 Lepodolite
10 Salt
20 Chalk -

Strong Lithia line in water — after boiling hard — Reboiled & still get Li line -

10 Lepodolite
10 Salt,

Strong Lithia line in water filtered there — no Lithia line on subtracting screen

This seems better than when lime is added -

After boiling more get Li line — but very faint & only at times

Note

31 Mlg/dl Oft in 250 cc water Seems the limit on direct Vision Spectroscope ~~see~~ ~~[illegible]~~ ~~faint~~ Cant say that I can see it, probably [illegible] good

64 Mlg is steady line faint Comparatively — $\frac{1}{4000}$ OK —

Very bright yellow white

#15 Fusion is
10 dependolate
30 Chalk
2 Salt,
} [illegible] fused Viscous

#14 is
10 Lepidolate
10 gypsum
3 Salt.
} fused

#10
is 10 dependolate
10 salt
20 Chalk.
} fused

#13 is
10 dep
10 Salt —
} were fused producing melted +

No 10 — Must fuse a longer time — Duplicate this fusing after reaching yellow white for 1 Hour —

Another Sample 2 hours

Final test of #10 diluted to 250cc much stronger than 1/2 gram & nearly if not as strong as Standard gram of Lithia, on 3 gr 6 oz

~~#10 — 3rd Exhaustion 12 hours on hot plate all night was dry at 10 am Sunday morning Got 30cc very bright Lithia Line strong showing that time must be allowed to get in the cleavage planes of the crystals even windows only scratches up not dissolved~~

No 10 — 3rd Exhaustion only very faint Li line, 12 hours on hot plate & dry in morning 10 am —

Dep is 100 mesh instead of 30 which this is & 24 hours boiling & this followed off & a 2nd water leach couple hours boil think will get it all —

Later taking some of the powder from the 4th Exhaustion & grinding on asbestos the Lithia line is strong showing that the ore has not been attacked enough to free the Li OH

#10 Continued

My impression is that No 10 formula with finer ore + longer fusion, + regrinding fusion very fine as we did #10 after it came out of crucible that we will get most of the Li out

The question is - is time necessary

#13. The residue after 3rd Extraction has some Lithia showing Lep not powdered fine enough + not heated long enough to penetrate fully there some of the Lepidolite,

250 CC - Dont show more than 500 mlg - not near as good as No 10 -

Evidently time of 10 minutes

15 - 2nd Extraction

Very faint Li Line with liquid 50 cc — powder gives rather strong line.

Say equal to 250 mlg Lith in 256 — again shows lepidolite not fine enough & not heated long enough —

250 cc less & think than standard test of 500 mlg around this —

Note Standard 1 gm Lithia 250 cc
adding phos soda no precip on
boiling large precip —
delta 500 cc Lithia sol —
250 cc dont show precip
probably because the amount
that would precip is soluble
in 250 CC — ~~[illegible]~~
~~[illegible]~~,

Phenomenon →

#14

It is not so strong
as 500 mlg 256 standard
Lithia Slice in Ranchu —
Must Crush are finer
& free longer — residue faint
to nothing —

So far #10 Best, in fact
very good, c-

Note in test tube with Na phosphate
No 10 no precip
13. a little
15 Considerable
14 bulky precip

Evidently none of the processes up to now gives good yields —

Have written up several more.

Note, I took a little lepidolite powdered it fine + put it on platina lid + brought it up to yellow on tripod. 15 minutes then under micro it showed that 50% of the mica had whitened + puffed then heated 15 min more + additional ones puffed, after 35 to 50 minutes 99% had puffed but a few showed central specks clear + not puffed. This shows that if kept for couple hours at a yellow all will puff. The leaves separate. I also notice that when I put the red hot Platina suddenly in cold water it broke a large part up, then I ground this wet up Martar + went almost to a jelly. Undoubtedly this

treatment is good for any
kind of a process, Especially
if acids are to be used or
even fusions & this
preliminary treatment is
not expensive — of the ore &
has about 3/4 lead mica & 1/4
clear quartz like material
beautifully drawn powder.

Lepidolite, ground 30 mesh
ignited yellow till caked together
while hot - taken out & crushed
finer. Then ignited again
thrown in hot water, & ground
again —

200 cc H_2O + 10 grams dep
with 10 or 15 cc Hydrochloric
gives strong line, better than
500 ~~milligrams~~ grams of LiOH +
not quite so much as
1 gram LiOH, acidified by HCl —
250 water —

2nd extraction by HCl —
250 cc water, same no graphite
that 62 Wiley standard LiOH.

Above is wrong as to double
ignition. There was only 1
ignition & final grinding
otherwise OK

I now start on the double
ignition

Experiment X

Lepidolite about 50 grams thru 30 mesh ignited alone till sticks together Crucible thrown in water till cooled — some stuff went into large jar water perhaps some Li Oh dissolved in water which I collected Then the water filtered to get floating stuff & balance removed from Crucible 95% stayed in Crucible — The whole reground very quite fine powdery 150 @ 180 mesh — The Reignited thrown in water same way Collected + reground —

This is what I am using

X dep

10 Grams above in 100 cc water Only —

in 2 hours nothing no line put in about 5 grams NaCl

This brings out some lithia not strong yet —

10 Lep X

5 cc Conc Sulphuric —
50 cc water 17th 1130 am

Residue much less in bulk than HCl Extraction
no mica visible, Think the now mica transparent is all attacked as see no pieces 1/2 white + 1/2 clear,
think about 750 mlg in 10 Lep.
it lasts longer than HCl —

Easy to filter clear solution
weight of dried residue 2.920.
Now Evaporating to dryness will drive off acid after drying + weighing + then reweigh

10 Lep X —

50 cc H_2O - 5 cc HCl — 17th 10 30 am
hot plate,

Residue much more than sulphuric Extraction,
no mica is visible, even opal micaceous
Notice a few pieces of clear stuff 1/4 white other part clear but no 1/10 the number there was before we double ignited + double ground

Think Li have checks more bright than with Sulphuric but dont think it lasts as long + Dont think there is more than 750 Mlg Li in the 10 Lep HCl extraction.

Note. Its almost impossible to filter the solution clear after 3 or 4 times its still milky Weight of dried residue is 4.540 grams some lost in filtering.
Now Evaporating to dryness will weigh the HCl to be driven off

~~Reweighed the [illegible] then driven off [illegible]~~
Reweighed after washing + deleting the 250 cc [illegible] grams

OOE

~~Lithia line about equal to 125 mg standard solution 150 cc ... showed equal to 500 @ 750 cc standard — white ... the alpha.~~

The original 001 250 cc Evap dacon showed 1.484 gm this is amount dissolved by the 5 cc HCl —

Li line about same as 500 mg in 250 cc standard — when dry residue dissolved — 250 cc water —

The 4.540 all night in more HCl — evap to dry HCl driven off — then 50 cc water Cant get the Li line ~~at first after~~ 1/2 hour get it strong — it seems to take time to get out of cleavage or porous stuff —

Hence Li + 1.484 Residue — total 6 gm 4 lost in alkali + residue on filter paper see further along

I note that in the first trial of HCl on 10 drop ~~and~~ not evaporated the 250 cc Evap to dry that just before dry it was a jelly — probably silica

10 Lep. 80 000 in a ton Sulphuric would dissolve 7 out of 10 grammes

or 1244 lbs this is entirely too much — 1333 lbs lime acid used —

Must now try the minimum H_2SO_4 that will remove the most of the Lithia —

With HCl 5 cc. 911 lbs of ore would be dissolved + 911 lbs HCl would be required — Must also determine minimum HCl that will do the trick

10 Lep X double ignition
HCl 5cc 250cc water
Continued –

Think 5cc to 10 grms too much – Think 3 cc will do just as well –

The residue will be when 5 cc HCl to 10 Lep used 300 lbs –
in this is the alkalies + other stuff –

Sulphuric Acid & 10 Lep X
5 cc H_2SO_4 Conc is out of the question it dissolves a considerable amount of alumina + frees the silica but only little of any Silica dis in the 250 cc water like the 10 grms was treated with its all alumina & phosphates not by ~~so~~ far HCl but by far

Note –

Just heated piece lepidolite size of first yellow & phosphorus with Conc water – it turned white & crystals friable ~~but~~ crushed with hand into coarse powder, this will make it easy to grind, but dont think mica broke ~~are separated~~ This will be done after 2 month grinding & ignition, possibly a longer grinding & another ignition –

Cold water containing X
Lep 1 gram passing CO_2
for long time don't appear
to dissolve any Li —

I will now try SO_2 in water
which is stronger acid —

In morning found it dried on hot plate its been on all day Tuesday night 6 pm added 5 cc Conc HCl & filled up to 250 cc to make up some ½" evaporation —

Lyons Calculation shows that to get all alkalies out alone require 8 to 10 cc HCl 37% + 3 cc was totally insufficient

One 10 gm Lep exhausted with fresh acid each time, for many hours 2nd showed strong Li Line after several hours. The 3rd exhaustion had its residue ground extremely fine in agate mortar & put in 10 cc acid after several hours Li Line very weak & difficult to get now on for 4th Ex 5 cc HCl — all night will test for Lime with less water

No 1 —

Pink Lep - Ignite 100 mesh then ignited, quenched water 10 grams 250 cc water 3 cc Conc HCl —

Left warm on hot plate all night but not in actual contact covered over with watch glass —

This Lep is very much richer than the White Lep been using Examined in glass before HCl — Mass all white +, whole looks good for leaching on gas 2 hours, off at 6 pm 18th until morning —
Line about like 500 Mg standard But its pink + much richer in Li

No 2.

Pink Lep- fine + then ignited
Repowdered, - in SO_2 15 hours
Cold, Sol still very acid even
after boiling 2 hours -
(not strong boil) - its strong
in Li line with 250 cc

Residue in about 15 cc water
+ 5 @ 10 cc HCl acting on it for
5 min gives strong Li line -

No 3 Cold 250 cc H_2O in flask

3 cc Conc HCl — see addition of HCl at 6 pm —

10 grm Lep ignited in muffle

Quenched — Reground & ignited Cold leach

in small Crucible; dep wont high grade only slight pink —

12 Noon 19th. Its redish

probably from iron,

Under Micro, scarcely any mica — what there is is opal — considerable quantity of clear quartz like particles, but all are finer than usual — There is abscent the white porous non micaceous pieces seen before —

at 6 pm put in 3 more cc HCl

Total 6 cc HCl —

Dec 23 1 cc deducted to 15 cc line

OK but V Very faint

It was at the end slightly acid

Think I used about 10 grams Caustic Lime - Sol Hot,

filters well, quickly, but requires lots washing on filter to get rid of Lithia Lime, 6 times so far & lime still there but weak now

After all out of lime & filtrate gave no lime, I put some of the Mud on wire pad & got brilliant Lithia line. Can it be some Li is combined stuff with ore

Now break filter & wash mud thru into 2 Casseroles to heat the water to boil & refilter to see if Can't get Li out,

I filtered the big sol which had lime thrown down by Oxalic filters clean & rapid, There is not much lime in it —

see new book

No 4 Leach solutions 75 C on butter dish -

Dup of #3 same stuff

3 cc HCl — see addition 3 cc at 6 pm -

Thru to be warm —

12 Noon 19

at 6 pm put in 3 cc more HCl Total 6 cc HCl 9 cc HCl

Dec 23. 9 45 am 1 cc diluted to 15 cc get lime weak but steady & a little better than No 5 at 5 cc dilution.

Off. 2.45 pm Dec 27 - I used say 20 cc of the liquid prepared

Now whole ore & all treated with WV & Caustic Lime & filtered -

at 11 am - 26th found only dull Lithia line showing not much more has come out but should say the Oxad must be very fine & well ignited & 10 days given it warm -
(note) sure that a large amount Li want locked up in the particles yet, but they look light & act light and are snow white,

The weight of Residue is 7.904 - I lost probably 100 to 250 Milg on paper

Treated residue till Dec 30th with H_2SO_4 - then filtered, washed residue water, filtered, then put this with 1st bag of filtrate & treated all with lime filtered & treated with oxalic when free of Ca. filtered this is a conclusion for Group to Group = note oxalic made after lime was filtered off a precip but Sol acid, also Ox of Na both acid to the H_2SO_4 I used must have something else

Had oxalic in case Back which gave surprise precip

No 5

Oxadumane, yellow in Muffle then in Aqua-Ground, ignited in small Crucible filtered & decanted, dried & reground - not very fine - 5 cc HCl. 250 cc aq

On warm - 19th 4 pm -

at 6 pm can only get a trace of Lithia line,

All night 11 30 am 20th only get a Li line like a little better than 62 standard

4.30 pm 21 Dec I notice that the Li line is very bright - time to see element as well as heat possibly weeks would extract it all -

at 9.30 am - Dec 23 - Took 1 cc diluted it to 5 cc line full & dull.

Dec 24 5 pm Removed solution by filtering & put back residue for further leaching with 5 cc Conc HCl - on plate at

#6

Dup of 5 —

Cold

250 cc ag

All night — 1130 am — Cant get the Li Dine —

4.30 pm 21 Dec Lithia line is only about 250 mg standard — not anywhere near as bright as Dup hot,

Dec 23 — 11 am — 1 cc detubed with 5 cc Cant see Lithia line

11 am 26th Dec
Only shows rather a dark line
rather quickly fading —
looks as if most Li was out of
ore,

The 10cc HCl brot out considerable
more Li, & the residue is now
small, with very little Li when
powder is ignited —

No 7

Ambiguite, Muffle to yellow
Quenched, ground, ignited in
small Crucible, yellow,
Quenched, Reground
10 grms 10 HCl Conc 250 ccs
to go on hot plate on
butter dish —

19th 4 20 pm

In ½ hour got Excessively strong Lithium
Line

Dec 23 10 am — 1 cc diluted to
50 cc get Line faint butter
butter Uses spectrum 5.

at 3 pm + no Li coming out in water
I put in 200 cc water & 10 cc Conc
HCl — to go on Hot — Dec 23 3 pm

8 Dup of 7
but heated Cold
19- 4 20 pm
250 cc aq

Dec 23, 11 am 1 cc diluted to
50 cc line & think is even
stronger than Hot Dup
No 7 - apparently showing
no advantage of heating
+ is probably not a porous
leach from fine holes but a
Dissolve - or an extremely porous
condition due to ignition.

time is an element as the spongy particles must have extremely fine pores + as acid is very dilute it will take a long time to come + go — should say in practice <u>One week</u>.

The only question seems to be ignition furnace + time of leaching — for Lepidolite,

I ignited lepidolite chunk weighing about a lb in muffle to fuse yellow — then suddenly threw in water, it turned white + was friable but only to pass about 1/16 — by grinding these very fine in agate mortar it is seen that all the iron component + would not be attacked by acid except outside surfaces —

It evidently wants a 2nd ignition in very fine powder + quenching + even this will not ~~ex~~ solution all perhaps 2/3 will solution —

Possibly a 3rd ignition might ~~satisfa~~ spongy the whole. But if all can be ground fine enough to float off think 2nd ignition will do it, + even then it should be leached + the leached stuff regrounded finer + relleached

According to Lotts figures 2000 lbs 4% Li metal in a Lepidolite ore + 10% K metal will require 1640 lbs of 37% HCl acid to turn the 2 alkalis into chlorides

10 gram sample needs 820 cc aprox of HCl - to Exhaust it, supposing ore had 4% Li metal + 10% K metal -

Dec 20 - 1918

Pink - high grade Lepidolite double ignition -

250 cc Aq + 5 cc HCl - strong
2nd Exhaustion 250 cc fresh water 5 cc HCl - This was strong Li line.
3rd Exhaustion: the residue was reground very fine in agate mortar + 5 cc fresh HCl + 250 cc fresh water used - This also gave a good Li line - The put in to 250 cc fresh Aq + 5 cc HCl all night on hot plate, - Lithia line a little weaker than 62 Milg standard, probably 50 Milg left

By proper ignitions + flow like grinding - could get nearly all out with 10 HCl - leaving very little in Residue

Weight of residue including a little I got off the filter paper which was light fluffy stuff —

Weight of residue in Casserole 7.100 —

Weight from filter paper — 2.308

Total 9.408

Some Lugl chloride must have been taken up — as we only have taken out .592 mlg.

It doesn't seem possible that the extraction was all dict

It made an error, I put the Casserole residue + the filter paper residue together + he made it.

Real weight of Total Residue

7.662

Extracted 2.338

I notice when I heated residue after weighing to weed it into gas come off - probably not dry

I ignited the entire residue from the residue weight of 7.662 grams and in minutes crucible weighing 2.250 it broke in 500 HCl for 1½ hours filtered & filtered — put in 500 HCl for 1½ hours filtered & filtered — 2.250 — 2.250 — 2.250

No. 9 –

Lepidolite, fair grade not pink
ignited in muffle yellow, quenched, powdered not.
Extra fine. Then ignited in small crucible till
it tends to stick on top, lid fritted white, quenched
ground fine –

10 grammes – 8 cc conc HCl commercial
put in flask, on hot plate on butter dish
200 CC water, poured in HCl, then ore
then water, On 2 pm. 20 Dec Hot

~~at Dec 23 10 am Test [illegible]~~
~~[illegible] Alle better~~
~~Then Specimen No 5~~

Leo

No 10 Dup of 9 but cold
Leach — 200cc aqr & HCl -

Note =

No 11

Is the same Lep at 9+10 but in this case the ignition in the small crucible was higher so it had passed viscous thick state & got solid so iron rod wouldn't move it Repowdered fine —

200 cc aq 8 HCl Conc 10 Lepidolite to go in a flask on hot plate on butter dish —

20th Dec 2.20 pm —
put ore in conc HCl + aq aqua regia —
~~Dec~~ -

The ore on watch glass seem to be more disintegrated than 9+10 fusion — but there is a large amount of transparent there yet, entirely too much to get good extraction;

Dec 21 4.30 pm I tipped flask & lost about 10 cc aqua but none of the ore —

No 12 Dup of 11 but
Cold beach,

Dec 20 2 30 pm

fused good, puffed up bad

13. Chunk Lamited & powdered + the powder fused

200 CC water

20 grms of following Mixture

25 grms pacor Lepid olite (white)
25 " Carb Soda —

fused till semi liquid ..
ground up — powdered fine

Now leaching in water only

Dec 24 9 30 am on heater plate
Butter dish —

off - Dec 28 - washed (1e) filtered
Used big beaker full as of
Li_2CO_3 it wanted acc. demand

then recalculated the reaction
with HCl - filtered

14 No 14 —

Dup of 13 Except,

The Mix is 25 Lep (white)
& 12 Carbonate Soda —

of this 20 grams of mixture is
used, 200 cc Water

Dec 24 — 9 30 am on plate with
butter dish —

Strong Li line so salt does not extract all — perhaps I should have not used acid but just water & give it time to percolate out of pores —

This Experiment should be tried again —

I notice the mud dont give as line but if wet with Barl gives good line.
Li appears locked up with something Leaving by Barl —

I added water after exhausting all I could with alcohol & dissolved the salt then added excess alcohol to precip salt after filtering dont get much salt
think it dissolves somewhat in alcohol

Lost

15

Lep Chunk ignited in Muffle & quenched powdered — then fused Lep 1700 grs

fused Mix 25 grms white Lepidolite
25 " Salt.

fuses ok, hard to get out of Crucible

Of this took 20 grms finely powdered 200 CC water only
Equals 10 grms Lep
Wgc 24 on butler dish
hot plate 9.45 am —

filtrate brot to dry
mitchsalt Crystals and ground up & treated with alcohol — different to extract all the LiCl from it as some must be in the Crystals of salt ground it stice Clamos out of 3rd Extractions with alcohol after grinding —
treated Residue with acid and filtrate from this gives

#16 — Lump Lep white, ignited in muffle bright yellow quenched, Powdered,

25 g. Lep
50 g Sulphate Soda -
fused together — Then powdered fine

20 grms of this used for Leach

200 cc water only

Dec 24 9.45 am in water only On Buchner funnel on hot plate —

✓ Trichlorohydrin fair
✓ Orthocresol good
✓ Monobromo Naphthalin faint
✓ Monochlorophenol fair
✓ Methylene Chloride good
~~Alcohol Butyrate Strong line in flame~~
✓ Benzene Anthracic faint (doubtful)
✓ Guaicol Bright line in flame
✓ Paramidophenolat faint
✓ Amyloxyd Butyric good when flaming
✓ Butylic Ether. Brilliant line
This is fine solvent
✓ Methylal faint
✓ Anisol Aldehyde faint
✓ Metaldehyde good line
✓ Methyl Ethyl Ketone good line
✓ Monochloro ortho phenol faint
✓ Isobutylbromide "
✓ Methylphenylamine "
✓ Trichresol CP. fair
✓ Eosigsaure Anhydride ? Brilliant

Many of these should be followed & verified as LiCl got on side of test tube —

~~Organic Solvents~~

The following organic solutions are discolors of LiCl

✓ Amyl Acetate good 0.18 in 100 W
✓ Amyl Alcohol Very good — S Sol W
✓ Acetone Very good "
✓ Pyridine Very good ?
✓ Benzaldehyde
✓ Nitro Toluol — good — doubtfull it was in H_2O_2 VV Little Sol W
✓ Monoaethyl ortho Toluidin fair " "
✓ Dichlorhydrin Pretty good — 1.1 in 100
✓ Paraphenetidin. faint after flaming cool
S.W. Meta Toluidin good " "
✓ Methylbenz Aniline Very good
✓ Perchlorethylen faint after burning off
✓ Creosote oil " " "
✓ Orthophenetidin faint "
✓ Eucolaptol faint
✓ Picoline Very good
✓ Butylic Alcohol Very good
✓ Methylene Chloride good — ? ?
✓ Benzol Chloride faint
✓ Phenylhydrazin good
✓ Dichlorobenzol good

These fine 180 mesh plates shrunk considerable —

Diameters of 4 discs	Thick
2.214	.446
2.230	.448
2.229	.448
2.238	.451

The Die is 2.287 —

Shrunk 58/1000. in Diameter

Battery 13

100 CuO & 200 mesh lead glass
250 mg q - Soda glass — Baked yellow
CuO then 180 mesh
Run at 600 ma per disc
rate ordinary limit
Ordinary temperature

Dec 26 11 ~~[illegible]~~ Am —

14 Battery

~~Dup of 13~~ —

In Dec 26, 11 am

Battery 15

Dup of 13 Except
run at 200 Milliamp
rate in Ice Chest
on Dec 26. 11 am

Battery 16

Dup of 13
Except discharg at
600 Milamp in
ice chest
in Dec 26 11 am

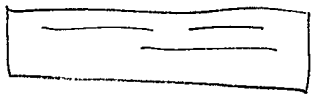
17-+18

Cut by red heat
on $CuSO_4$ 4 Pb glass
1 gram Dry Soda

2 Discs

Did not shrink
any in muffle —

19 - precipitated
CuO - heated just
dull red
4 grms lead glass
1 " Dry Soda

Shrunk much
from 2.25
to 2.10 -

Cracked
1 time